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3-(4-Bromophenyl)-2-methylacrylic acid

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.087
Data-to-parameter ratio = 13.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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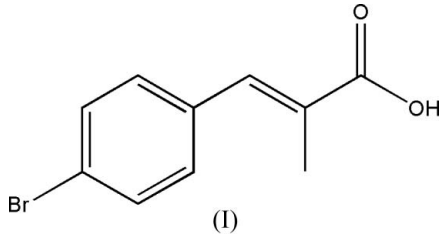
The title compound, $C_{10}H_9BrO_2$, displays an E configuration about the C=C double bond. In the crystal structure, symmetry-related molecules are linked by O—H···O hydrogen bonds, forming centrosymmetric carboxylic acid dimers.

Comment

Cinnamic acid and its derivatives have been reported to possess a variety of pharmacological properties, including hepatoprotective (Perez-Alvarez *et al.*, 2001), antimalarial (Wiesner *et al.*, 2001), antioxidant (Natella *et al.*, 1999) and antihyperglycemic activities (Liu *et al.*, 1999). They constitute a large family of organic acids that have antibacterial, antifungal and antiparasitic activities, as well as antitumour and chemopreventive properties (Liu *et al.*, 1995). The molecular structure of compound (I) is shown in Fig. 1, and selected bond distances and angles are given in Table 1. The C7—C8 bond length of 1.349 (3) Å shows it to have double bond character, and the configuration about this bond is *E*. The bond lengths within the benzene ring range from 1.389 (4) to 1.403 (3) Å, typical of aromatic character (Allen *et al.*, 1987). Intermolecular hydrogen bonding between carboxyl groups in the crystal structure of (I) results in the formation of dimers. These dimers are further linked by intermolecular C—H···O bonds, forming a chain-like structure. (Table 2 and Fig. 2).



(I)

Experimental

Compound (I) was synthesized according to a reported method (Gensler & Berman, 1958). A mixture of 4-bromobenzaldehyde (1.85 g, 10 mmol), methylmalonic acid (2.36 g, 20 mmol) and piperidine (1.98 ml, 20 mmol) in pyridine solvent (12.5 ml) was heated on a steam bath for 24 h. The reaction mixture was cooled and added to a mixture of 25 ml of concentrated HCl and 50 g of ice. The precipitate formed in the acidified mixture was filtered off and washed with ice-cold water. The product was recrystallized from ethanol. The yield was 90%.

Crystal data

$C_{10}H_9BrO_2$
M_r = 241.08
Triclinic, $P\overline{1}$
a = 7.3202 (11) Å
b = 8.1460 (13) Å
c = 9.0526 (14) Å
α = 114.431 (2)°
β = 107.444 (2)°
γ = 92.065 (2)°
V = 460.85 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.42 mm^{-1}
T = 100 (1) K
0.44 × 0.17 × 0.14 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.243, T_{max} = 0.539
3745 measured reflections
2129 independent reflections
1932 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 1.03
2129 reflections
154 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 1.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br—C3	1.897 (3)	O2—C9	1.232 (3)
O1—C9	1.321 (3)	C7—C8	1.349 (3)
Br—C3—C4	118.1 (2)	O2—C9—C8	121.4 (2)
Br—C3—C2	120.49 (19)	O1—C9—O2	122.4 (3)
O1—C9—C8	116.2 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···O2^{i}	0.81 (5)	1.83 (5)	2.646 (3)	177 (7)
C2—H2···O2^{ii}	1.03 (3)	2.46 (3)	3.447 (3)	160 (3)
C7—H7···O1	0.99 (3)	2.36 (4)	2.729 (4)	101 (2)

Symmetry codes: (i) $-x+3, -y, -z+1$; (ii) $x-1, y, z-1$.

All H atoms were located in a difference Fourier map and were refined with isotropic displacement parameters. The refined C—H distances are in the range 0.92 (4)–1.04 (4) Å. The highest residual density peak is located 1.0 Å from the Br atom.

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2006); data reduction: *SAINT-Plus*; program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLUTO* (Meetsma, 2007) and *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

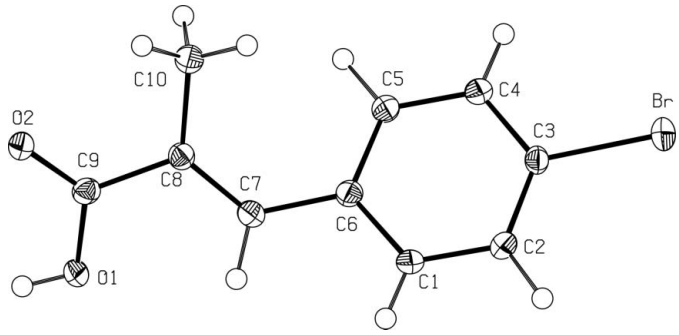


Figure 1
The molecular structure of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. H atoms are drawn with an arbitrary radius.

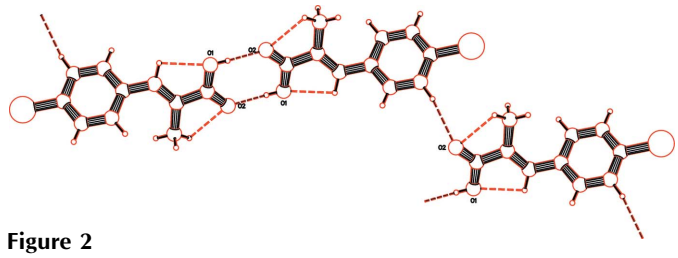


Figure 2
Perspective drawing of the dimer formed by O—H···O hydrogen bonds and the connection by C—H···O interactions to give a chain.

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supporting information

Acta Cryst. (2007). E**63**, o2174–o2175 [https://doi.org/10.1107/S1600536807014468]

3-(4-Bromophenyl)-2-methylacrylic acid

Niaz Muhammad, Zia-ur-Rehman, Saqib Ali and Auke Meetsma

3-(4-Bromophenyl)-2-methylacrylic acid

Crystal data

$C_{10}H_9BrO_2$
$M_r = 241.08$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3202$ (11) Å
$b = 8.1460$ (13) Å
$c = 9.0526$ (14) Å
$\alpha = 114.431$ (2)°
$\beta = 107.444$ (2)°
$\gamma = 92.065$ (2)°
$V = 460.85$ (12) Å^3
$Z = 2$
$F(000) = 240$

The final unit cell was obtained from the xyz centroids of 2400 reflections after integration using the SAINTPLUS software package (Bruker, 2006).
Reduced cell calculations did not indicate any higher metric lattice symmetry and examination of the final atomic coordinates of the structure did not yield extra symmetry elements (Spek, 1988; Le Page 1987, 1988)
Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.
Le Page, Y. (1988). *J. Appl. Cryst.* **21**, 983–984.
Spek, A. L. (1988). *J. Appl. Cryst.* **21**, 578–579.
$D_x = 1.737$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2400 reflections
$\theta = 2.8$–$28.8°$
$\mu = 4.42$ mm^{-1}
$T = 100$ K
Block, colourless
0.44 × 0.17 × 0.14 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine focus sealed Siemens Mo tube
Parallel mounted graphite monochromator
Detector resolution: 66.06 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2006)

$T_{min} = 0.243$, $T_{max} = 0.539$
3745 measured reflections
2129 independent reflections
1932 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.8°$
$h = -7 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.03$
2129 reflections
154 parameters

0 restraints
Primary atom site location: heavy-atom method
Secondary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 1.14$ e Å^{-3}
$\Delta\rho_{min} = -0.78$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.19136 (4)	0.40680 (3)	0.08590 (3)	0.0260 (1)
O1	1.2601 (3)	−0.0520 (2)	0.3293 (2)	0.0202 (5)
O2	1.4011 (3)	0.1606 (2)	0.6036 (2)	0.0207 (5)
C1	0.6965 (4)	0.1901 (3)	0.0822 (3)	0.0169 (6)
C2	0.5274 (4)	0.2569 (3)	0.0335 (3)	0.0182 (7)
C3	0.4232 (4)	0.3156 (3)	0.1457 (3)	0.0164 (6)
C4	0.4841 (4)	0.3079 (3)	0.3037 (3)	0.0177 (7)
C5	0.6543 (4)	0.2430 (3)	0.3508 (3)	0.0175 (7)
C6	0.7655 (4)	0.1859 (3)	0.2426 (3)	0.0165 (6)
C7	0.9484 (4)	0.1197 (3)	0.2892 (3)	0.0171 (6)
C8	1.0833 (3)	0.1785 (3)	0.4482 (3)	0.0161 (6)
C9	1.2610 (4)	0.0947 (3)	0.4671 (3)	0.0172 (7)
C10	1.0810 (4)	0.3320 (3)	0.6127 (3)	0.0198 (7)
H1	0.766 (5)	0.143 (4)	0.009 (4)	0.022 (7)*
H2	0.493 (5)	0.263 (4)	−0.083 (4)	0.030 (8)*
H4	0.410 (5)	0.350 (4)	0.377 (4)	0.023 (7)*
H5	0.695 (5)	0.230 (5)	0.465 (5)	0.040 (9)*
H7	0.978 (5)	0.030 (4)	0.189 (4)	0.023 (8)*
H10	1.217 (4)	0.410 (4)	0.685 (3)	0.012 (6)*
H10′	1.028 (4)	0.283 (4)	0.680 (4)	0.022 (7)*
H10"	0.996 (5)	0.410 (4)	0.593 (4)	0.027 (8)*
H11	1.363 (6)	−0.087 (5)	0.352 (5)	0.036 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0216 (2)	0.0353 (2)	0.0243 (2)	0.0151 (1)	0.0071 (1)	0.0159 (1)
O1	0.0195 (10)	0.0212 (8)	0.0172 (8)	0.0103 (7)	0.0055 (7)	0.0061 (7)
O2	0.0182 (9)	0.0237 (8)	0.0163 (8)	0.0076 (7)	0.0037 (7)	0.0065 (7)
C1	0.0177 (12)	0.0172 (10)	0.0136 (10)	0.0038 (9)	0.0056 (10)	0.0047 (8)
C2	0.0188 (13)	0.0204 (10)	0.0146 (11)	0.0046 (10)	0.0041 (10)	0.0082 (9)
C3	0.0132 (12)	0.0171 (10)	0.0168 (11)	0.0036 (9)	0.0032 (9)	0.0070 (8)
C4	0.0175 (13)	0.0185 (10)	0.0148 (11)	0.0033 (9)	0.0051 (10)	0.0057 (9)

C5	0.0184 (12)	0.0198 (11)	0.0164 (11)	0.0045 (9)	0.0065 (9)	0.0095 (9)
C6	0.0181 (12)	0.0146 (10)	0.0153 (10)	0.0038 (9)	0.0049 (9)	0.0057 (8)
C7	0.0173 (12)	0.0171 (10)	0.0177 (11)	0.0046 (9)	0.0074 (10)	0.0074 (9)
C8	0.0165 (12)	0.0171 (10)	0.0170 (11)	0.0042 (9)	0.0064 (9)	0.0091 (9)
C9	0.0200 (13)	0.0175 (10)	0.0159 (11)	0.0044 (9)	0.0068 (10)	0.0087 (9)
C10	0.0205 (13)	0.0182 (10)	0.0178 (11)	0.0051 (10)	0.0054 (10)	0.0062 (9)

Geometric parameters (Å, º)

Br—C3	1.897 (3)	C7—C8	1.349 (3)
O1—C9	1.321 (3)	C8—C9	1.488 (4)
O2—C9	1.232 (3)	C8—C10	1.503 (3)
O1—H11	0.81 (5)	C1—H1	0.92 (4)
C1—C6	1.403 (3)	C2—H2	1.03 (3)
C1—C2	1.394 (4)	C4—H4	0.95 (4)
C2—C3	1.390 (4)	C5—H5	1.04 (4)
C3—C4	1.394 (3)	C7—H7	0.99 (3)
C4—C5	1.389 (4)	C10—H10	1.02 (3)
C5—C6	1.402 (4)	C10—H10′	1.01 (3)
C6—C7	1.471 (4)	C10—H10"	0.94 (4)
Br···Br^{i}	3.4958 (7)	C9···H4^{xi}	2.82 (4)
Br···H10′^{ii}	3.19 (3)	C9···H11^{iii}	2.77 (4)
O1···O2^{iii}	2.646 (3)	C10···H5	2.67 (4)
O2···O1^{iii}	2.646 (3)	H1···H7	2.40 (5)
O2···C9^{iii}	3.327 (4)	H1···O1^{v}	2.78 (3)
O2···O2^{iii}	3.210 (3)	H2···O2^{ii}	2.46 (3)
O1···H5^{iv}	2.75 (4)	H4···C8^{ix}	3.09 (4)
O1···H10′^{iv}	2.73 (3)	H4···C9^{ix}	2.82 (4)
O1···H1^{v}	2.78 (3)	H4···C4^{viii}	2.93 (3)
O1···H7	2.36 (4)	H4···H4^{viii}	2.49 (5)
O2···H2^{vi}	2.46 (3)	H5···C8	2.93 (4)
O2···H10	2.46 (3)	H5···C10	2.67 (4)
O2···H11^{iii}	1.83 (5)	H5···H10′	2.51 (5)
C1···C2^{vii}	3.525 (4)	H5···H10"	2.28 (5)
C2···C1^{vii}	3.525 (4)	H5···O1^{iv}	2.75 (4)
C2···C6^{vii}	3.554 (4)	H5···C9^{iv}	2.96 (4)
C4···C4^{viii}	3.568 (4)	H5···H11^{iv}	2.50 (6)
C4···C9^{ix}	3.353 (4)	H7···O1	2.36 (4)
C5···C10	3.143 (4)	H7···H1	2.40 (5)
C6···C10^{x}	3.598 (4)	H10···O2	2.46 (3)
C6···C2^{vii}	3.554 (4)	H10···C1^{x}	2.97 (3)
C9···O2^{iii}	3.327 (4)	H10···C2^{x}	2.90 (3)
C9···C4^{xi}	3.353 (4)	H10···C3^{x}	2.93 (3)
C10···C6^{x}	3.598 (4)	H10···C4^{x}	3.06 (3)
C10···C5	3.143 (4)	H10···C6^{x}	3.07 (3)
C1···H10^{x}	2.97 (3)	H10′···Br^{vi}	3.19 (3)
C2···H10^{x}	2.90 (3)	H10′···H5	2.51 (5)

C3···H10^{x}	2.93 (3)	H10′···O1^{iv}	2.73 (3)
C4···H10^{x}	3.06 (3)	H10"···C5	2.63 (3)
C4···H4^{viii}	2.93 (3)	H10"···C6	2.83 (3)
C5···H10"	2.63 (3)	H10"···H5	2.28 (5)
C6···H10"	2.83 (3)	H11···O2^{iii}	1.83 (5)
C6···H10^{x}	3.07 (3)	H11···C9^{iii}	2.77 (4)
C8···H5	2.93 (4)	H11···H5^{iv}	2.50 (6)
C8···H4^{xi}	3.09 (4)	H11···H11^{iii}	2.58 (6)
C9···H5^{iv}	2.96 (4)		

C9—O1—H11	109 (3)	O1—C9—O2	122.4 (3)
C2—C1—C6	121.6 (3)	C2—C1—H1	122 (2)
C1—C2—C3	118.6 (2)	C6—C1—H1	117 (2)
Br—C3—C4	118.1 (2)	C1—C2—H2	116 (2)
C2—C3—C4	121.4 (3)	C3—C2—H2	125 (2)
Br—C3—C2	120.49 (19)	C3—C4—H4	119 (2)
C3—C4—C5	119.1 (3)	C5—C4—H4	122 (2)
C4—C5—C6	121.2 (2)	C4—C5—H5	119 (2)
C1—C6—C7	119.1 (2)	C6—C5—H5	120 (2)
C5—C6—C7	122.8 (2)	C6—C7—H7	114 (2)
C1—C6—C5	118.1 (3)	C8—C7—H7	118 (2)
C6—C7—C8	127.3 (2)	C8—C10—H10	110.5 (15)
C7—C8—C10	126.6 (2)	C8—C10—H10′	110.9 (19)
C9—C8—C10	114.5 (2)	C8—C10—H10"	112.8 (19)
C7—C8—C9	118.8 (2)	H10—C10—H10′	111 (2)
O1—C9—C8	116.2 (2)	H10—C10—H10"	108 (3)
O2—C9—C8	121.4 (2)	H10′—C10—H10"	103 (3)

C6—C1—C2—C3	−1.8 (4)	C4—C5—C6—C7	178.8 (3)
C2—C1—C6—C5	3.0 (4)	C1—C6—C7—C8	146.2 (3)
C2—C1—C6—C7	−177.9 (3)	C5—C6—C7—C8	−34.8 (4)
C1—C2—C3—Br	−179.7 (2)	C6—C7—C8—C9	−179.0 (3)
C1—C2—C3—C4	−0.3 (4)	C6—C7—C8—C10	−3.2 (5)
Br—C3—C4—C5	−179.5 (2)	C7—C8—C9—O1	−10.0 (4)
C2—C3—C4—C5	1.1 (4)	C7—C8—C9—O2	169.3 (3)
C3—C4—C5—C6	0.2 (4)	C10—C8—C9—O1	173.7 (2)
C4—C5—C6—C1	−2.2 (4)	C10—C8—C9—O2	−7.1 (4)

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) *x*−1, *y*, *z*−1; (iii) −*x*+3, −*y*, −*z*+1; (iv) −*x*+2, −*y*, −*z*+1; (v) −*x*+2, −*y*, −*z*; (vi) *x*+1, *y*, *z*+1; (vii) −*x*+1, −*y*, −*z*; (viii) −*x*+1, −*y*+1, −*z*+1; (ix) *x*−1, *y*, *z*; (x) −*x*+2, −*y*+1, −*z*+1; (xi) *x*+1, *y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···O2^{iii}	0.81 (5)	1.83 (5)	2.646 (3)	177 (7)
C2—H2···O2^{ii}	1.03 (3)	2.46 (3)	3.447 (3)	160 (3)
C7—H7···O1	0.99 (3)	2.36 (4)	2.729 (4)	101 (2)

Symmetry codes: (ii) *x*−1, *y*, *z*−1; (iii) −*x*+3, −*y*, −*z*+1.